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MORPHOLOGICAL AND FLOW CHARACTERISTICS OF SYNTHESIZED POLYSTYRENE COMPOSITES

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ABSTRACT

A variety of polymers are used in engineering and medical applications. Polystyrene is one of the most important commodity polymer widely used in technical applications. It has applications in household goods, packaging, automobiles and other engineeringapplications. Four fillers calcium carbonate, graphite, mica and talcum powder of micrometer size in the form of powder with threelevel of concentration 1%, 2%, 3% by weight were synthesized with polystyrene. The composites were produced by in-situ polymerization method and the samples were prepared by hand operated injection moulding machine. The melt flow index was performed to study the effects of fillers and their concentrations on the flow properties of composites. The morphological analysis by SEM and EDX were also performed. The particle size was found to be varying from 0.2 µm to 0.5 µm.Two-way ANOVA using "Minitab 15" software was used to see the contributions and significance of the input parameters on the output parameters. The addition of fillers are found to be significant while concentrations of fillers insignificant at 5% level of significance.

Keywords: Composites, Polystyrene, Flow characteristics, Morphologyand ANOVA.

1. INTRODUCTION

Composites are the combinations of two or more materials in which areinforcing material is embedded in a matrix in a controlled manner to obtain a new material having distinct properties.Varieties of polymers for composites are thermoplastic polymers, thermosetting polymers, elastomers, and their blends.Conventionally, there are three methods to synthesize polymer composites. They are in-situ polymerization, melt intercalation and solution methods.[1] Studied the synthesis and characterization of polystyrene clay nanocompositesby melt intercalation, in-situ polymerization and masterbatch methods. At less than 1 % clay, in-situ formed nanocomposites

showed the best improvement in tensile, flexural, impact strength and Young's modulus. The maximum improvement was 88.5 %, obtained at 0.73 wt. % organoclay in the in-situ formed material.[2]Studied the characterizations of expanded graphite/polymer composites prepared by in situ polymerization. Microscopic results disclosed that the expanded graphite has a legume-like and honeycomb sub-structure survived after hot-pressing, resulting in a graphite network penetrating through the entire composite body, which produces a composite with excellent electrical conductivity.[3] Studied the effect of talcum filler content on the mechanical properties of polypropylene composites. They examined that the increase in filler content lead to an increase in the strength of the composite material with a simultaneous decrease in the fracture toughness and the increase in tensile strength were from 15 to 25% and the maximum tensile strength at break was found to be 22 MPa. [4]Studied the effects of mica, with varying concentration5 to 40 weight % of mica prepared by twin screw extrusion, on mechanical, thermal, electrical, rheological and morphological properties of polyester thermoplastic elastomer and depicted that the flexural strength and modulus increased with mica concentration, whereas tensile strength decreased at higher concentrations. Morphological studies revealed that there is a good dispersion of filler in the polymer matrix at lower concentrations.[5] Explored the nano-calcium carbonate ($CaCO_3$)/polystyrene core-shell nanoparticle. All composites were prepared individually by incorporating nano-$CaCO_3$/PS hybrid nanoparticles and bare nano-$CaCO_3$ with 0.10–5.0 wt% on BrabenderPlastograph. It was shown that rheological, thermal, mechanical and morphological properties were improved as hybrid nano-$CaCO_3$/PS particles reinforced in high impact polystyrene (HIPS) matrix.

1.1 Objectiveof the Study

In this work, the effects of graphite powder, mica powder, calcium carbonate and talcum powder and their concentrations1%, 2% and 3% by weight on melt flow index and SEM/EDX of synthesized polystyrene polymer matrix composites produced through in situ polymerization method is explored and the properties of the output parameters of the composites are analyzed by a software "Minitab 15" using two-way analysis of variance (ANOVA).

2. DESIGN OFEXPERIMENT

2.1 Input and Output Variables

Table1 Input and Output Variables

Input Variables		Output Variable	
Filler Materials	Graphite Powder, Mica Powder, Talcum Powder, $CaCO_3$	Morphological Analysis	SEM, EDS
Filler Concentration	1%, 2%, 3% By Weight	Flow Characteristics	Melt Flow Index (MFI)
Stirrer Speed	800±50 rpm		

Table 2 Quantity of Additives used

Mass of Styrene (in grams)	% ByWeight	Mass of Additives (in grams)
590.85	1	5.9085
590.85	2	11.817
590.85	3	17.7255

2.2 Polystyrene

Polystyrene (PS) belongs to the group of standard thermoplastics that includes polyethylene, polypropylene and polyvinylchloride. Polystyrene is used in an extremely wide range of applications [6]. Principal characteristics include transparency, ease of coloring and processing and low cost [7]. Mechanical andrheological behavior of polystyrene is determined by its average molecular weight; the strength improves with increasing chain length but the melt viscosity increases as well making processing difficult [6].

2.3Properties of Fillers

Calcium carbonate ($CaCO_3$) is a white powder and colorless crystal. Its melting point is 825°C and specific gravity as 2.83. It is stable and has negligible water solubility. Graphite is good conductor of electricity having specific gravity 2.2. Structure of graphite has hexagonal rings and stable forms of carbon. Micahas density 2.9 $g/cm^3$and is transparent, flexible, elastic,and chemicallyinert and caneasilyresist actions of heat, light, water, oil, solvents, and alakalies. It is strongly absorbent to Ultraviolet radiations, prevents penetration of destructive Sun-rays. Pulverized talcum powder has wide industrial applications as filler in rubber, textile, plastic, linoleum, asbestos products, polishes and soaps; as a loading agent for all kind of papers; as a carrier of insecticidal and for coating calcium ammonium fertiliser.

2.4 In-Situ Polymerization Method

In this method, the layered silicate is swollen within the liquid monomer. Polymerization of the monomer occurs in the interlayer of the clay mineral, resulting in an expanded interlayer distance. Polymerization can be initiated by heat or a suitable initiator [8].

3. EXPERIMENTATION

3.1Experimental Procedures in the Preparation of Polymer Composites

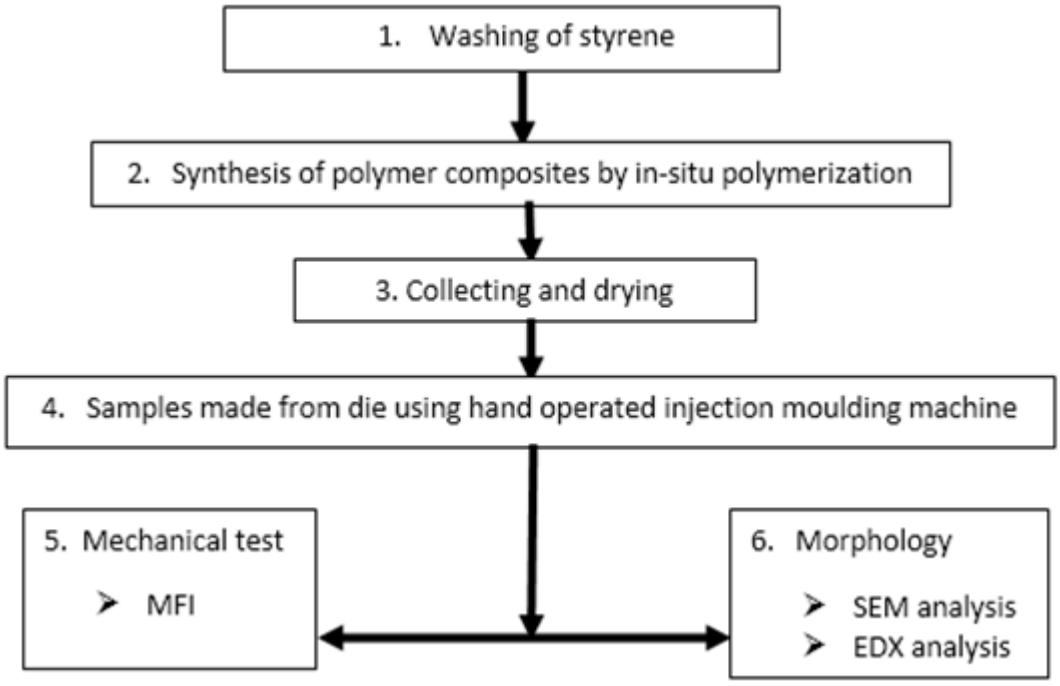

Figure 1 Experimental procedures in the preparation of polymer composites

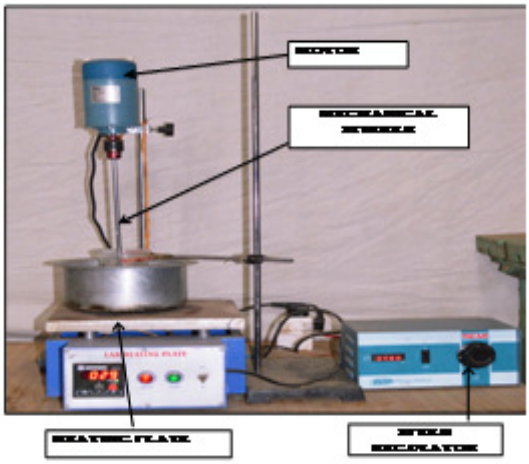

Figure 2 Experimental setup

Figure 3 Working on experimental setup

3.2 Synthesis, Collecting and drying of composites

Styrene monomer containing the desired amount of additives was mechanically mixed by stirrer for 30 minutes at room temperature in order to obtain better dispersion. The benzoyl peroxide in calculated quantity was then added to the mixture to initiate the polymerization which took place at 90°C at speed of about 800±50 rpm of stirrer in each case for approximately three hours till the monomer solution becomes viscous i.e.; completion of the reaction. After completion of the reaction add 250 ml of methanol to separate the viscous composites from the beaker.The composites are collected into a plate and left the sample to dry for 48 hours. Fig. 4 showsdifferent synthesized composites.

(a) **(b)** **(c)** **(d)** **(e)**

Figure4 (a) Pure polystyrene, Polystyrene composites with(b) Mica (c) Calcium Carbonate (d) Talcum powder (e) Graphite

3.3 Sample Preparation

The composites obtained at the end of polymerization were kept in an oven at 120°C for 2-3 hours to complete polymerization and remove the remaining moisture and liquid styrene. Then the samples were prepared by hand operated injection moulding machine and fabricated die.In injection molding the material is heated until it melts. The melt is then injected into die and the product is ejected. During molding, barrel andmold temperatures were maintained at 210°C and 30°C respectively for the preparation of each sample.Die was fabricated according to size of the specimen required ASTM D 638 'Type I'.Materials for die and ejector pins were mild steel and silver steel respectively.

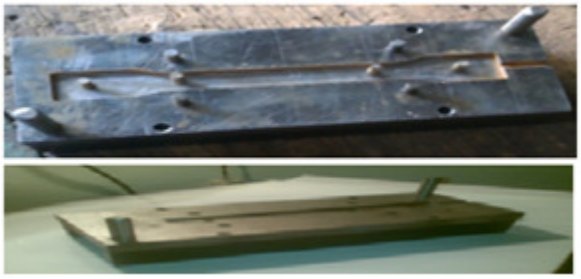

Figure 5 Fabricated adjustable die

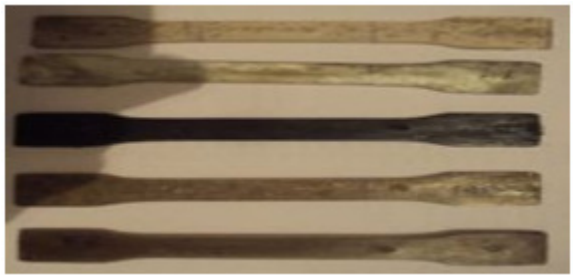

Figure 6 Prepared samples

4. RESULTS

4.1 Micrographs by Scanning Electron Microscope and Energy Dispersive X-Ray Spectroscopy

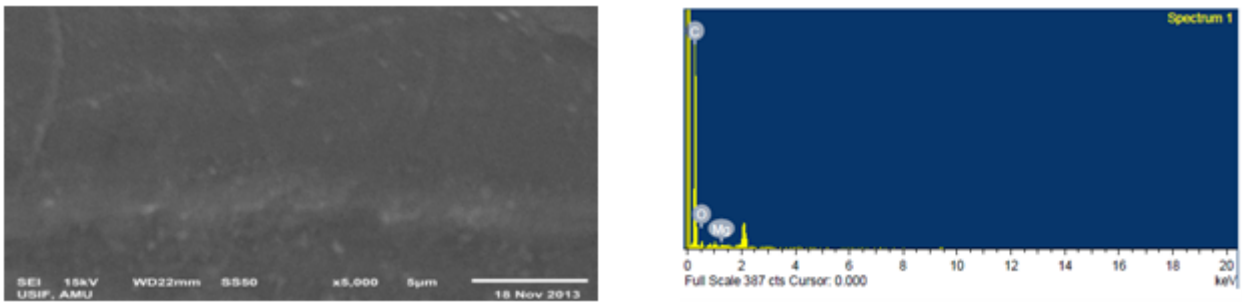

Figure 7 Micrographs by SEM and EDX of synthesized pure polystyrene

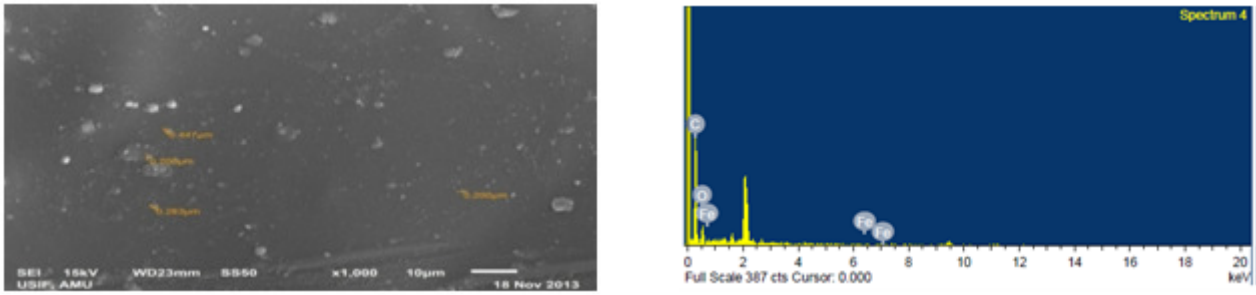

Figure 8 Micrographs bySEM and EDX of synthesized polystyrene graphite composite

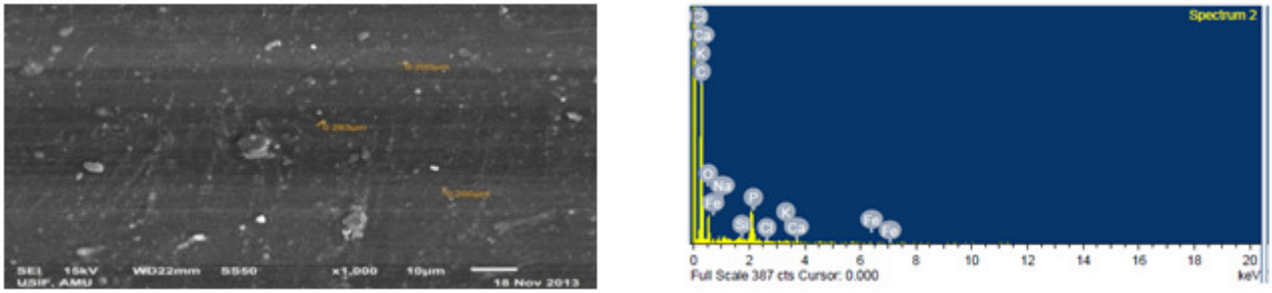

Figure 9 Micrographs by SEM and EDX of synthesized polystyrene mica composite

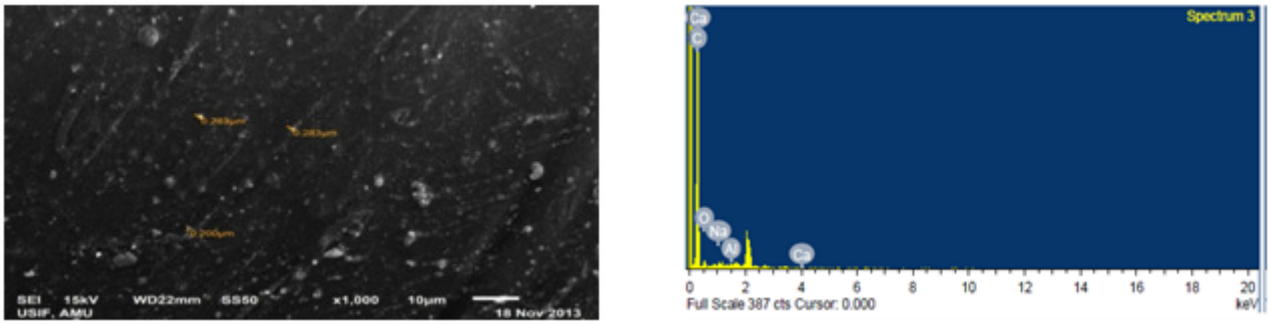

Figure 10 Micrographs bySEM and EDX of synthesized polystyrene $CaCO_3$ composite

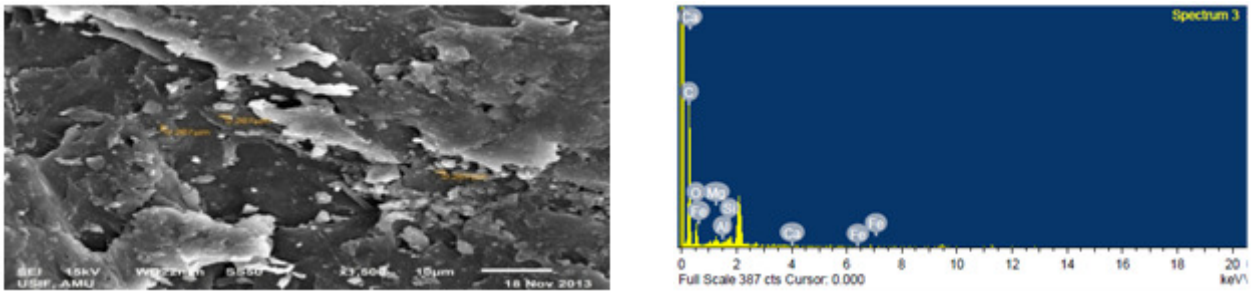

Figure 11 Micrographs bySEM and EDX of synthesized polystyrene talcum composite

4.2 Melt Flow Index

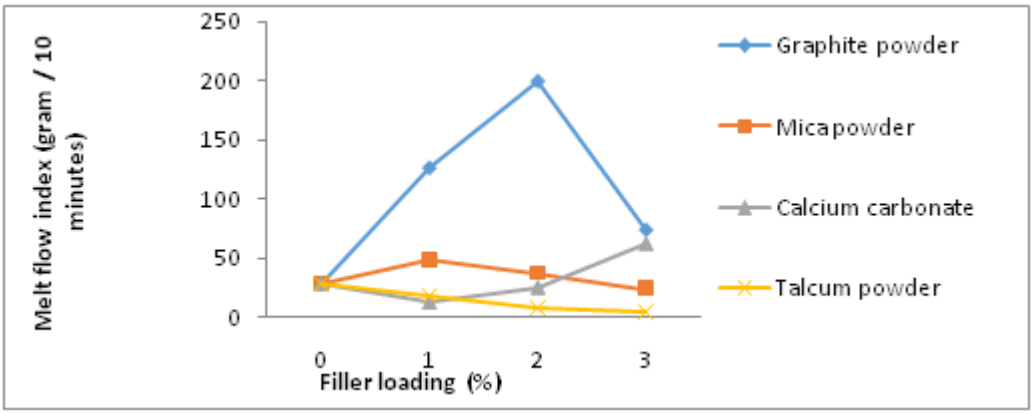

Figure 12 Effect of fillers on Melt Flow Index of synthesized polystyrene composites

Table 3ANOVA Table for MFI

Source	DF	SS	MS	F-value	P-value	% Contribution
Filler loading	3	3241.2	1080.39	0.65	0.604	8.41
Fillers	3	20258.3	6752.75	4.05	0.045	52.59
Error	9	15019.6	1668.84			39.00
Total	15	38519.0				100

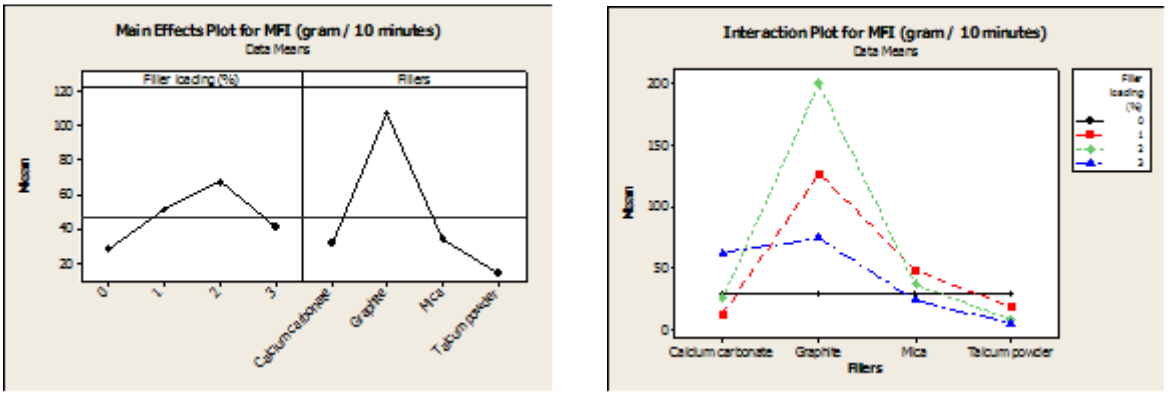

Figure 13 Response graphs by ANOVA for Melt Flow Index

5. DISCUSSIONS

5.1 Scanning Electron Microscope and Energy Dispersive X-Ray Spectroscopy

Fig.7 to 11show the micrographs of different composites made byin-situ polymerization techniqueby adding different fillers. In order to observe the effect of addition of fillers on the morphology of the prepared surfaces of the composites were analysed by means of scanning electron microscopy (SEM) mainly focusing on the homogeneity. The main purpose of micrographsis to see the degree of dispersion and filler matrix interaction. Micrographs were taken between X1000 and X5000 magnifications.Fig.7 shows micrograph of pure polystyrene which is a brittle polymer having straight propagation lines rather than zigzagged or tortuous lines. In such polymer, because of the homogeneous structure, there are not any barriers to stop the crack propagation. The peaks micrographs were taken by energy dispersive X-ray spectroscopy (EDX), show the constituents present in the PS and their composites. It is depicted thatthe particle size of synthesized PS-graphite composite was varying such as 0.200µm, 0.283 µm, 0.447 µm and uniform dispersion of graphite particles in the PS matrix. This is due to the stirring of the constituents present in the composites.The particle sizes of synthesized pure PS- mica, $CaCO_3$and talcum powder composites were found to be 0.200 µm, 0.283 µm and 0.267 µm respectively. The dispersion of mica particles seems to be good with agglomeration at some places in the PS matrix while dispersion of $CaCO_3$ is very good and accurate in the PS matrix showing homogeneity. The degree of dispersion of talcum powder in PS matrix is better as compared to other additives, although showing agglomeration at some places in the PS matrix (Fig.11) because the particles of the talcum powder were embedded in the matrix. This is due to the better wettability between the constituents of the composites. Therefore, the mechanical properties are improved. Through the examination of the SEMmicrographs, they are not quite distinguishable from each other. For more accurate and precise analysis of the morphology, Transmission Electron Microscopy (TEM) analysis can be performed.

5.2 Melt Flow Index

Fig. 12 shows the effects of fillers and their concentrations on melt flow index of synthesized polystyrene composites. Melt flow index (MFI) test was performed to check the flow characteristics of the synthesized PS composites. The polymers having high MFI value indicate low viscosity and low molecular weight. So the polymer composites were synthesized in order to optimize the MFI value.The value of MFI of pure PS is found to be 28.28. It is depicted that as the filler percentage increases the MFI increase and then decreases in case of PS-graphite and mica powder composites. The maximum increase is 605% and 69.66% in MFI in case of PS-graphite and mica composites respectively, which shows very high flowability and very low viscosity. Therefore, the mechanical properties are not improved as much as others composites. For PS-$CaCO_3$ and talcum powder

composites, the maximumdecrease in MFI is 55.94% at 1% concentration and 84.15% at 3% concentration respectively. Therefore, in case of PS-talcum powder composite the highest increase in viscosity and poor flowability in the entire test is predicted. This is due to the better embedment of the particles in the matrix which can also be found by observing the micrographs as seen in the Fig. 10 and 11. These are the basic reasons for the improvement of mechanical properties. Table 3 shows analysis by two way ANOVA for MFI test. It is attributed that the percentage contributions of filler loading and fillers are 8.41% and 52.59% respectively while percentage contribution of errors is 39.00%. This error includes various factors such as experimental error, error due to thedesign of experiment, error during taking observations, etcetera. The filler concentration is insignificant and fillers are significant at 5% level significance because p-valuesare 0.604 and 0.045 respectively.

6. CONCLUSIONS

1. Polystyrene wassuccessfully synthesized with fillers by in-situ polymerization technique.
2. The better results were found in synthesized PS-talcum powder and PS-$CaCO_3$ composites because of good dispersion of talcum powder and $CaCO_3$ in PS matrix as seen by SEM analysis.
3. It is concluded that flowability of PS-graphite is very high i.e low viscosity as compared to other fillers and the flowability of PS-talcum powder composite is minimum, showing high viscosity.
4. The contribution of filler loading is 8.41% and the contribution of fillers is 52.59% in case of MFI test, analyzed by ANOVA. Also the filler loading was found to be insignificant and fillers are found to be significant at 5% level of significance.

REFERENCES

1. Ozden G, “Synthesis and Characterization of Polystyrene Clay Nanocomposites”. Polymer 52, 2004, pp.1847-1855.
2. Zheng G, Wu J, Wang W, Pan C, “Characterizations of expanded graphite/polymer composites prepared by in situ polymerization”, Carbon 42, 2004, pp.2839–2847.
3. LubomirLapcik, Jr., PavlinaJindrova, BarboraLapcikova, Richard Tamblyn Richard Greenwood, Neil Rowson Published online 26 August 2008 in Wiley InterScience, 2008.
4. Sreekanth M S, Bambole VA, Mhaske ST, Mahanwar PA. “Effect of Concentration of Mica on Properties of Polyester Thermoplastic Elastomer Composites”. Journal of Minerals & Materials Characterization & Engineering, Vol. 8, No.4, 2009, pp. 271-282.
5. Chatterjee A, Mishra S. “Nano-Calcium carbonate ($CaCO_3$)/Polystyrene (PS) core-shell nanoparticle: It’s effect on physical and mechanical properties of high impact polystyrene (HIPS)”. J Polym Res (2013) 20:249, DOI 10.1007/s10965-013-0249-7. 2013.
6. KrishnamoortiR.andYürekli K., “Rheology of polymer layered silicate nanocomposites”, Current Opinion in Colloid and Interface Science”, vol.6, 2001, pp.464-470.
7. “Ullmann’s Encyclopedia of Industrial Chemistry”, 5th edition, VCH Publishers, vol. A21, New York, 1992, pp.617-625.
8. KornmannX,” Synthesis and Characterization of Thermoset-Clay Nanocomposites”, Lulea University of Technology, Sweden, 2000.
9. D.Lingaraju, K.Srinivas, B.A.Ranganath, “Failure Analysis of Pin Loaded Glass Epoxy/Polystyrene Composite Plates” International Journal of Advanced Research in Engineering & Technology (IJARET), Volume 3, Issue 2, 2012, pp. 137 - 144, ISSN Print: 0976-6480, ISSN Online: 0976-6499